Chapter 14 - **SAMPLE ENVIRONMENTS**

A number of sample environments are available for SANS measurements. These consist in standard cells and cell holders that can be heated (up to 300 ^{o}C) using heating cartridges or cooled (down to 0 ^{o}C) using circulating baths. Between 0 ^{o}C and 10 ^{o}C (depending on the ambient dew point), the sample chamber must be evacuated and/or filled with inert gas (nitrogen or helium) in order to avoid condensation on cooling blocks. A host of special sample environments are also available at the NIST CNR. These include in-situ pressure cells, in-situ (Couette or plate/plate) shear cells as well as rheometers, electromagnets (up to 2 Teslas), a superconducting magnet (9 Teslas), low temperature closed cycle helium refrigerators for temperatures down to 5 K and even lower (below 1 K), and a furnace for temperatures up to 450 ^{o}C. Only a few highly-used pieces of equipment are described here.

1. STANDARD SAMPLE CELLS

SANS measurements involve a variety of different cells. The first type is the standard off-the-shelf "banjo cell" owing to its characteristic shape. This type is used for photon scattering as well and has quartz windows (transparent to both neutrons and light). Their diameter is 2 cm and are characterized by a sample gap thickness of either 1 mm or 2 mm corresponding to a sample volume of 0.3 ml or 0.6 ml respectively. This type of cell is appropriate for liquid samples that can be handled using a syringe.

The second type of cell used for SANS has grown out of successive iterations at the NIST CNR. It is of the demountable type with titanium body and quartz windows. An inner spacer ring of thickness either 1 mm or 2 mm is part of the cell body and sets the sample gap. The sample thickness is uniform between the two quartz windows. Sealing is performed through back-up o-rings and tightening retainers on each side. This type of cell can handle liquids, gels, wafers and powders. Gel and powder samples are loaded from one side after tightening the retainer piece on the other side. Slightly larger volumes than for banjo cells are required.

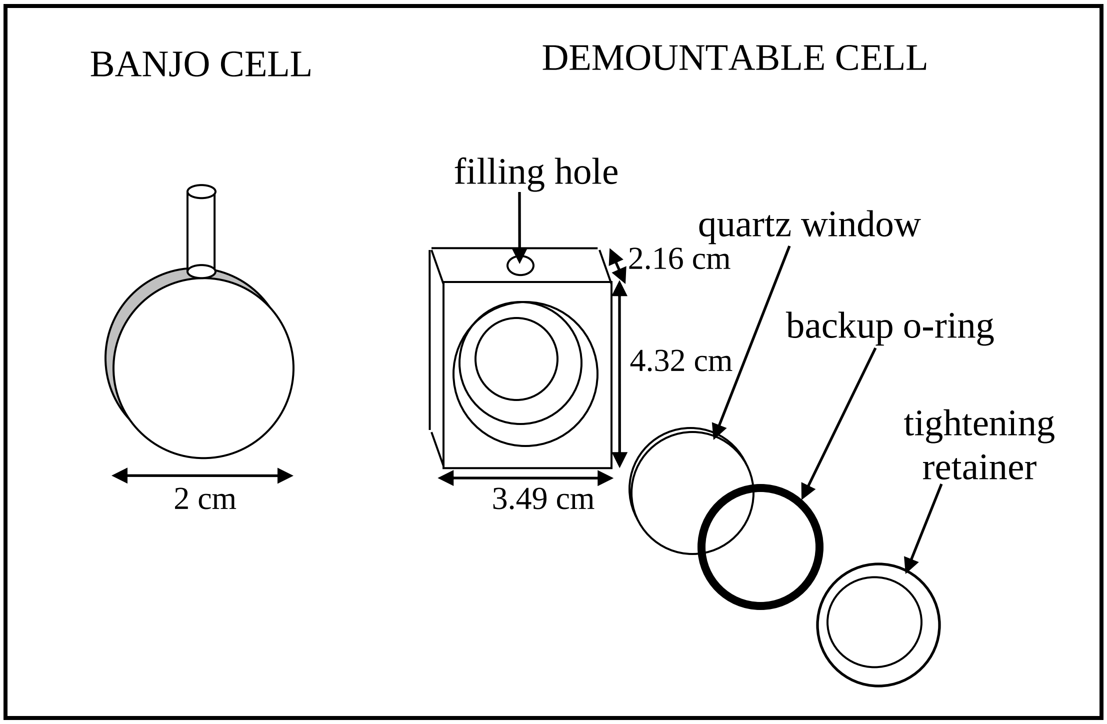

Figure 1: Sample holder cells used for SANS at the NIST CNR.

2. HEATING AND COOLING BLOCKS

A 7-position heating block made out of aluminum is used for temperature control. It uses two pieces of bakelite at the base to thermally decouple the main heated block from the other pieces in the sample chamber. This heating block controls temperature between ambient and 300 ^{o}C with a precision of less than 1 ^{o}C. The actual sample temperature lags slightly behind the block temperature. A resistance temperature detector (RTD) is used to monitor the block temperature.

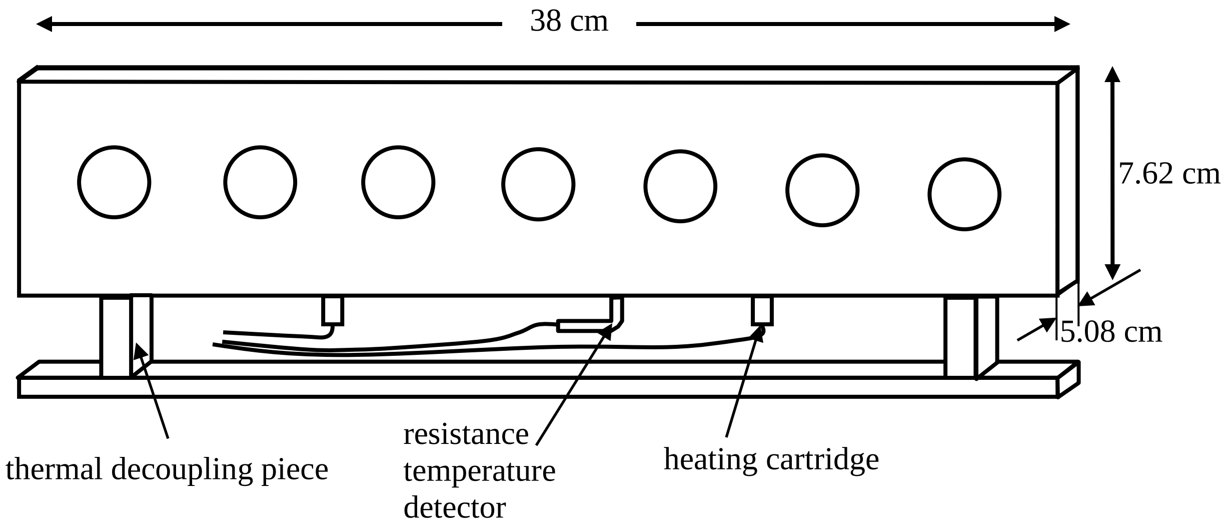

Figure 2: A 7-position heating block using heating cartridges and an RTD to monitor the temperature.

A similar (10-position) block uses flow of coolant instead (50 % water and 50 % antifreeze) to cool samples down to 0 ^{o}C. The heating range for that block is limited to 90 ^{o}C. Another heating/cooling block uses circulating silicon oil to reach an even wider temperature range (up to 150 ^{o}C).

3. THE PRESSURE CELL

Two generations of pressure cells for in-situ SANS measurements were designed to handle polymer samples. The polymer wafer is first melt pressed inside a metal ring to set a uniform thickness (of 1 mm). This produces a homogeneous clear sample. The right amount of sample (0.3 ml) is used in order to fill the available volume. This wafer is then transferred to the middle of a confining o-ring. This "encapsulated" o-ring (flexible rubber for the inside and Teflon coating for the outside) transmits pressure from the pressurizing fluid (water in this case) to the sample. The sample is also confined between two sapphire windows with a 1 mm gap between them. The cell body is made out of Iconel metal (75 % nickel, 15 % chromium) which is good for its high corrosion resistance and tensile strength at high temperatures. The pressure cell is surrounded by a heating jacket using heating cartridges for temperatures from ambient to 160 ^{o}C. Another (cooling) jacket uses a circulating fluid to reach down to 10 ^{o}C. The second generation pressure cell can handle up to 3 kbar pressures.

The pressurizing system consists of a pressure pump, two remotely controlled valves, high pressure tubing, two gauges, etc. The pump and the two valves are computer controlled and use a feedback signal from the digital gauge. The pressuring system and the main SANS data acquisition system follow a handshake protocol through a two bit process (“acknowledge” and “release” lines). When using a liquid sample, a separator is inserted between the pump and the pressure cell. This consists of a cylindrical tube with a piston inside to separate out the pressurizing fluid from the liquid sample.

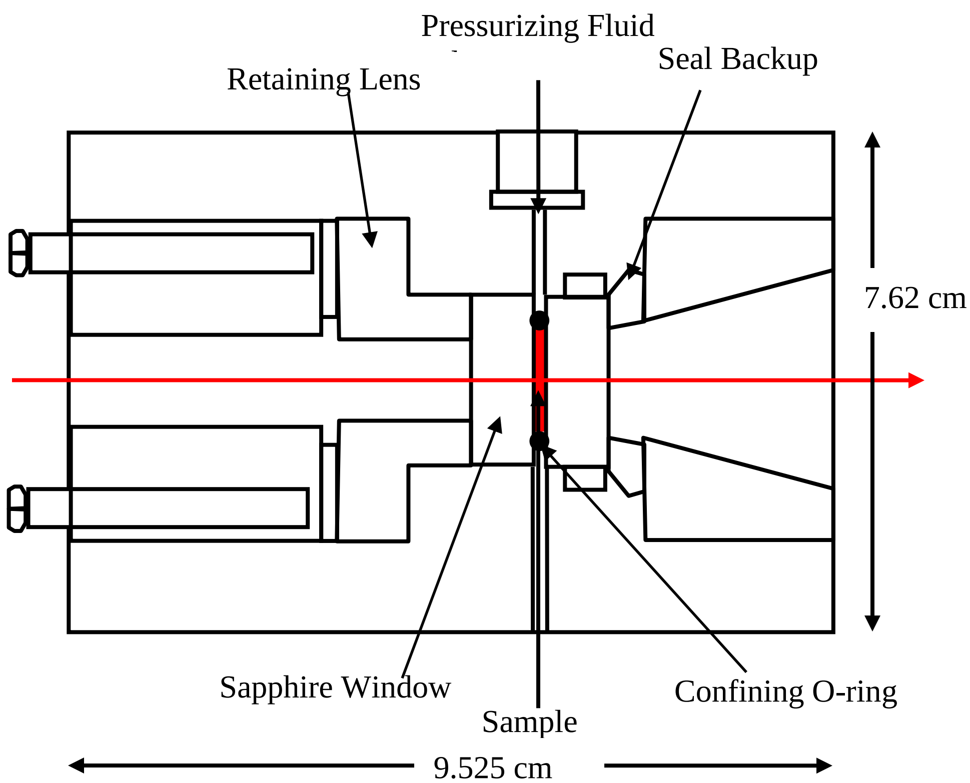

Figure 3: The first generation pressure cell for in-situ pressure measurements.

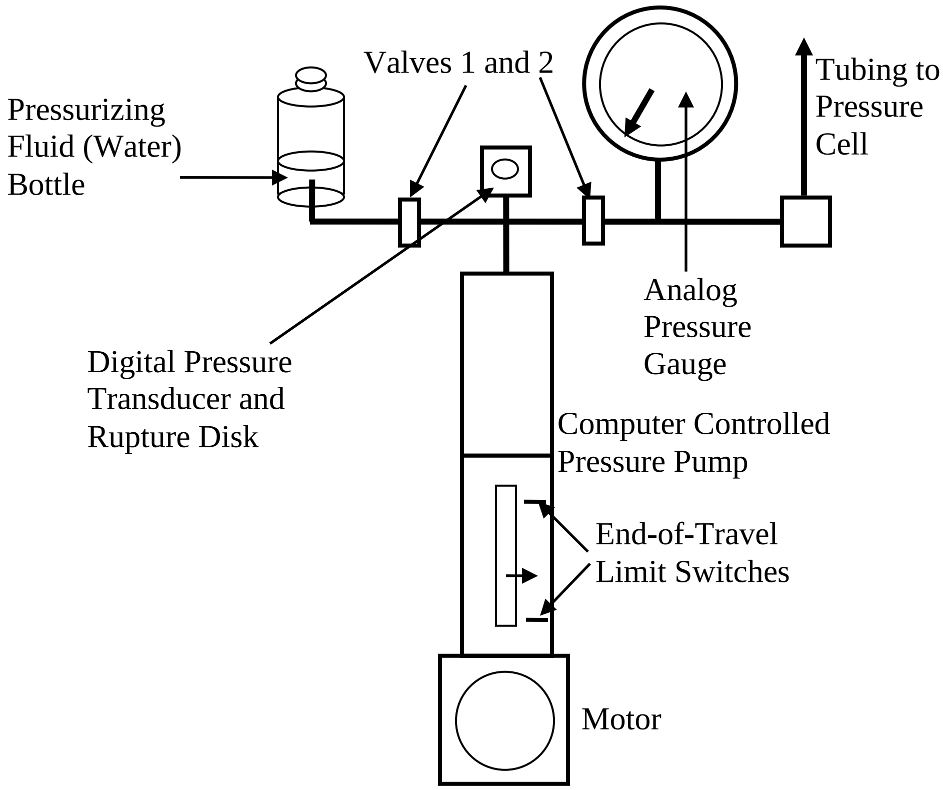

Figure 4: Schematic representation of the pressure cell system including the computer controlled pump and valves, the pressure transducer, the main gauge, and the various high pressure tubing. Note that the pressurizing fluid bottle is standing vertical (out of the page).

4. THE COUETTE SHEAR CELL

The Couette shear cell used for in-situ SANS measurements is a simple device for aligning samples. It consists of a stator which is lowered into a rotor cup to shear liquid samples within the 0.5 mm gap. Note that the neutron beam crosses the gap twice. The stator and the rotor are made out of quartz. The rotor base is made out of a material called Invar which has a thermal expansion coefficient similar to quartz. A set of x-y translation Verniers allows the precise alignment of the stator with respect to the rotor. The rotor cup takes about 12 ml. When the stator is lowered, the fluid sample level rises (in the gap between the stator and rotor) until it covers the neutron beam level.

The shear cell is used in one of two main geometries: (1) the radial mode whereby the 1.27 cm diameter neutron beam goes through the middle of the cell and (2) the tangential mode whereby a vertical slit (1.27 cm*0.3 mm) defines the neutron beam incident tangential to the cell.

This shear cell has been used for easily flowing liquids as well as for highly viscous fluids. Temperature control is performed using a circulating fluid. Coolant circulates inside the stator without getting in the neutron beam. Cell temperature can be controlled from 10 ^oC up to 90 ^oC. The shear cell is used in either the steady shear mode or the reciprocating shear (or jiggle) mode. Shear rates up to 5,000 Hz for are possible.

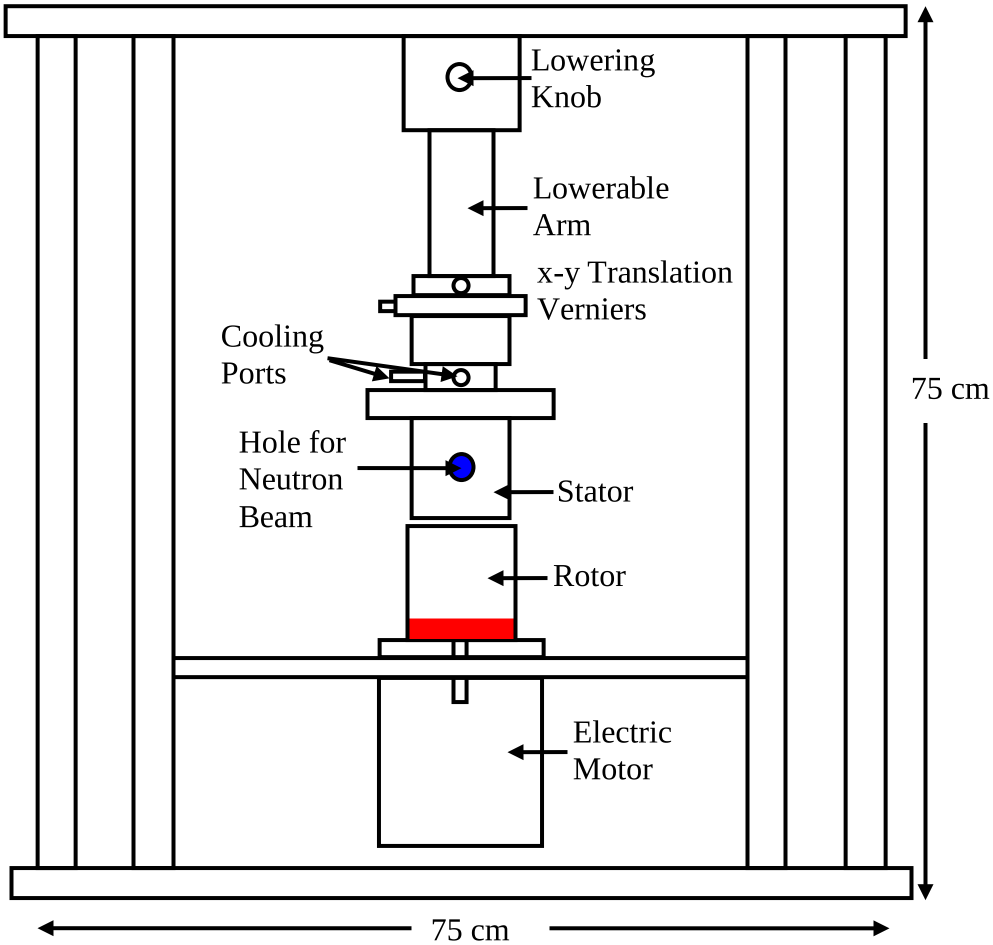

Figure 5: Schematic representation of the Couette shear cell setup with the stator in the raised position.

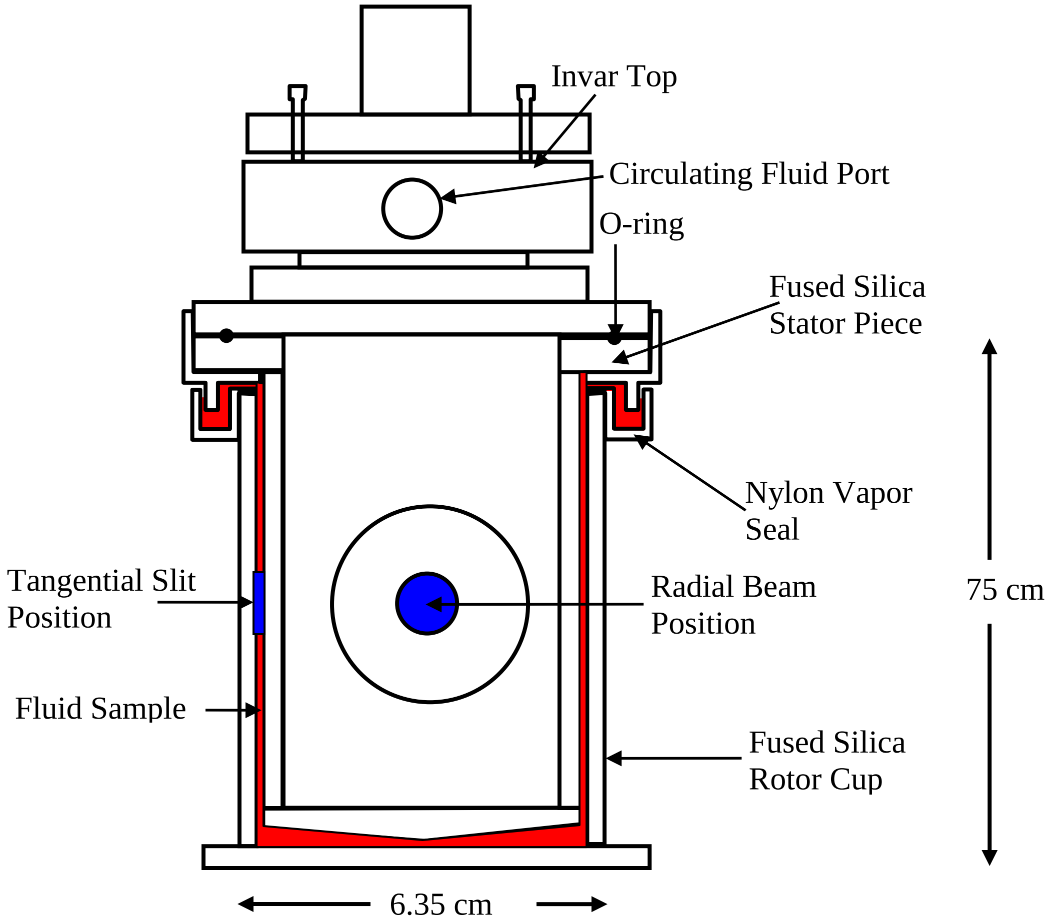

Figure 6: Schematics of the rotor and stator for the in-situ Couette shear cell. The neutron beam is perpendicular to the plane of this drawing.

5. THE PLATE/PLATE SHEAR CELL

A plate/plate shear cell is available at the NIST CNR for in-situ SANS measurements. This device was designed for investigations of oriented block copolymers. It consists of two arms; one fixed and one moving. The sample is melt pressed into a special (1 mm thick) holding cell which is mounted between the two arms. The fixed arm holding the sample is tightened in order to squeeze the sample between the two arms. The translation screw transforms the rotational motion (from the motor) into a translational motion (up and down). Two limit switches limit the travel range and therefore the strain. A strain of 100 % is obtained for a 1 mm travel.

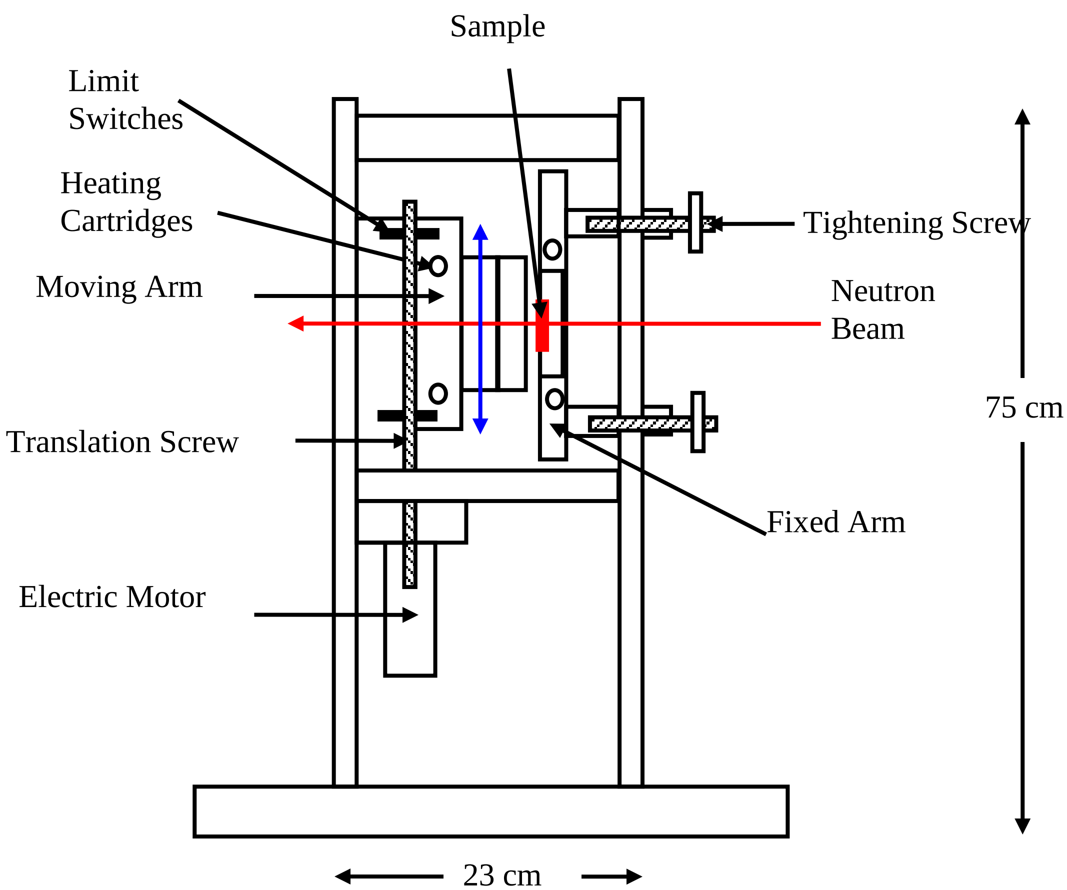

Figure 7: Schematics of the in-situ plate/plate shear cell.

6. OTHER SAMPLE ENVIRONMENTS

Other sample environments are available for in-situ SANS measurements at the NCNR. These include a couple pf rheometers for in-situ rheology. The shear cells described above can orient samples but cannot measure torque. The rheometers are standard equipment that were modified to allow a neutron beam to be incident on the sample and in-situ SANS measurements. The main modification consisted in raising the sample cup from its standard location (inside a temperature trough) to a higher (more accessible) location in the neutron path. Temperature control is performed through controlled air circulation.

Other pieces of equipment include electromagnets (up to 2 Teslas) and a superconducting magnet (up to 9 Teslas). A humidity chamber and a vapor cell allow sample humidity and vapor control. Other cells are available.

REFERENCES

G. Straty, "The NIST SANS in-situ Couette Shear Cell", Internal Report (1993)

B. Hammouda and S. Kline, "The NIST SANS in-situ Pressure Cell", Internal Reports (1995, 1998).

QUESTIONS

1. Why use quartz windows for sample cells?
2. What is the maximum reachable temperature for the heating block? What is the temperature range for the cooling block?
3. What are the units for ambient pressure?
4. Why doesn't water boil above 100 ^{o}C inside the pressure cell?
5. What is the characteristic of most SANS data with in-situ shear?

ANSWERS

1. Quartz windows are fairly transparent to neutrons and to light; quartz is less expensive than sapphire.
2. The heating block can reach up to 300 ^{o}C. The cooling block uses 50 % water and 50 % ethylene glycol (antifreeze) and can reach from 0 ^{o}C to 90 ^{o}C. The range between 0 ^{o}C and 10 ^{o}C is above the dew point (where water condensation occurs on windows). This range should be used only in inert (either nitrogen or helium) atmosphere to avoid water condensation.
3. Ambient pressure corresponds to 14.7 psi = 1 atm = 1 Bar. This is equivalent to 760 mm of mercury or 760 torr and converts to 101,325 Pa in SI units.
4. Water does not boil above 100 ^{o}C inside the pressure cell because a positive pressure (at least 100 psi) is always maintained.
5. Most SANS data with in-situ shear are characterized by anisotropic scattering with oriented contour maps.